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IS : 1448 [P : 119] - 1985
Adopted : 12 August 1985

(Reaffirmed 2002)

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 119]

AROMATIC TRACES IN LIGHT SATURATED HYDROCARBONS BY GAS CHROMATOGRAPHY

(First Reprint NOVEMBER 1998)
UDC 665·6/·7 : 543·544·25 : 547·31

Adapted from the Joint Publication No. ASTM D 2600-82/IP Designation 262/79

1. SCOPE

1.1 This method covers the determination of benzene, toluene, and C_8 aromatics in light saturated hydrocarbon samples. The method is limited by aromatic selectivity of the stationary liquid to samples containing *n*-decane as the highest boiling compound. (*see* Note).

NOTE — Some of the C_{10} naphthenes have boiling points below that of *n*-decane. Such compounds may be eluted after *n*-decane on the columns described in this method and may cause interference with the determination of benzene.

1.2 The method covers the concentration range from 0·005 to 1·00 volume percent of each C_6 to C_8 aromatic carbon number group.

1.3 Results are reported to two significant figures.

2. OUTLINE OF THE METHOD

2.1 The sample is introduced into a gas chromatographic column containing a strongly polar liquid phase. The polar phase has very little affinity

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for saturated and olefinic hydrocarbons, while exhibiting a pronounced selectivity for aromatics. This selectivity results in the elution of all saturated and olefinic hydrocarbons in the products described above prior to the elution of benzene. Either a thermal conductivity or flame ionization detector may be used. Calibration is performed by using synthetic blends of the aromatic compounds charged at the same constant volume as is used to analyse the sample. *n*-Undecane or *n*-dodecane is used as an internal standard.

3. SIGNIFICANCE

3.1 A knowledge of aromatic traces in light saturated hydrocarbons, such as light naphthas and solvents, is needed to assess purity of solvents and mee product specifications for trace aromatics as may be set by the consumers and regulatory agencies.

3.2 This method is also useful as a complementary method to IS : 1448 [P : 104]-1981*. It enables aromatics to be determined in naphthas and aviation gasolines at concentrations below the lower limit (1·0 volume percent) of method given in IS : 1448 [P : 104]-1981*.

4. APPARATUS

4.1 Chromatograph — Any chromatographic instrument may be used that has the following performance and characteristics.

4.1.1 *Sensitivity* — The system shall be capable of detecting 0·01 volume percent of any aromatic compound of interest. For calculation technique utilizing a recorder, the signal for that concentration shall be at least five chart divisions above the noise level on a zero to 100 scale chart. Noise leve shall be restricted to a maximum of one chart division. When electroni integration is employed, the signal for 0·01 percent concentration shall b at least twice the noise level.

4.1.2 *Sample Inlet System* — A sample inlet system that allows a constan volume of liquid sample to be injected.

4.1.2.1 If the apparatus is equipped with a sample splitter or if flame ionization detector is used, check the linearity of the splitter and detector. To do this prepare two standard samples containing 0·5 percen and 1·0 volume percent each of benzene, toluene, and one of the C_8 aromatics in *iso*-octane, as described in **7.1**. Using the same instrument conditions and sample sizes used for the method, inject a constant volume o each blend into the chromatograph. Measure the peak areas of the aromatic compounds and plot a graph of component concentration against pea area. This should be a linear relationship if the splitter and detector are working correctly. Make duplicate runs on each blend.

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*Aromatics in light naphthas and aviation gasolines by gas chromatography.

4.1.3 *Column* — Any column and conditions may be used provided, that under conditions of test, *n*-decane is eluted prior to benzene when tested as follows.

4.1.3.1 Prepare a blend of about 0·5 volume percent each of *n*-decane, internal standard, benzene, toluene, ethylbenzene in *iso*-octane.

4.1.3.2 Inject the blend into the chromatograph and record the chromatogram. The *n*-decane shall be eluted prior to benzene and the ratio of *A*/*B* shall not be less than 0·5 where *A* is the depth of the valley between the *n*-decane and benzene peaks and *B* is the height of the benzene peak (*see* Fig. 1).

NOTE 1 — Column materials and conditions which have been found suitable for this method are listed in Tables 1 and 2. (*See* Fig. 3 for a typical chromatogram for column 5 in Table 1.)

NOTE 2 — Some of the liquid phases used in this method, such as polyethylene glycol, will degenerate if heated in the presence of air or oxygen to a temperature above 50°C.

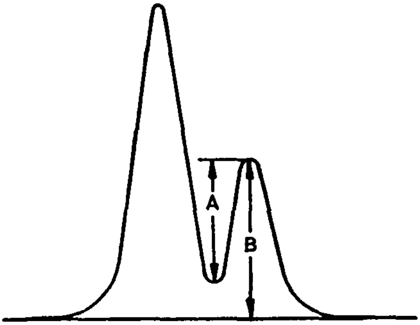

FIG. 1 ILLUSTRATION OF A/B RATIO

4.2 Strip Chart Recorder — A recording potentiometer with a full-scale deflection of 10 *m*v or less should be used. The full-scale response of the recorder should not exceed 2 seconds. If a manual method of integration (such as triangulation, paper cut-out or planimeter) is employed, the chart speed should be at least 1 500 mm/h in order to minimize errors in peak area measurement. This requirement is waived if a ball and disk or an electronic integrator is employed.

TABLE 1 TYPICAL COLUMNS AND CONDITIONS

(*Clause* 4.1.3.2 *and* 10.1.2)

COLUMN	1	2	3	4	5	6	7
Liquid phase	CEF	PEG 200	CEF	DEGS	TCEPE	TCEPE	DEGS
Length, m	4·5	2	5	3·1	4·5	31	3·7
Diameter, mm							
Inside	1·8	—	1·8	4·5	1·5	0·254	—
Outside	3·2	6·4	—	—	—	—	3·2
Mass, percent	17	30	20	20	10	—	20
Solid support	Chromosorb P	Chromosorb P	Chromosorb P	Chromosorb P	Chromosorb P	Capillary	Chromosorb P
Mesh	60 to 80	60 to 80	60 to 80	80 to 100	60 to 80	—	80 to 100
Treatment	AW	AW	AW	none	AW	none	AW, Sil
Inlet, °C	200	210	250	260	250	275	260
Detector, °C	200	155	250	200	175	250	240
Column, °C	115	95	90	100	115	95	65
Carrier gas	N_2	He	He	He	N_2	N_2	He
Flow rate, cm^3/*Min*	30	60	60	60	1	3	52
Detector	FI	TC	FI	FI	FI	FI	FI
Recorder, mV	5	1	1	1	10	1	1
Sample, µl	5	10	1	2	5	0·8	5
Split	9+1	—	—	—	100+1	100+1	—
Area	Tri	EI	DI	Tri Plan	EI	EI	Tri

Abbreviations used in Table:

DEGS — Diethylene glycol succinate
DI — Disk integrator
EI — Electronic integrator
FI — Flame ionization
He — Helium
PEG — Polyethylene glycol
Plan — Planimeter
N_2 — Nitrogen
CEF — N, N-Bis (2-cyanoethyl)-formamide
Sil — Silanized
TC — Thermal conductivity
TCEPE — Tetra-dyanoethylated pentaerythritrol
Tri — Triangulation
AW — Acid washed

TABLE 2 TYPICAL RETENTION TIMES, MINUTES

(*Clause* 4.1.3.2)

Column	1	2	3	4	5	6	7
Benzene	3·4	2·0	6·5	6·7	5·4	6·1	6·7
Toluene	4·4	3·2	9·0	10·3	7·8	7·0	10·3
Ethylbenzene	5·4	5·2	11·5	14·8	10·8	8·0	14·8
p-and *m*-Xylenes	5.8	—	12·5	—	11·4	8·5	—
o-Xylene	7·5	6·8	17·0	16·1	14·5	10·0	—
n-Undecane	3·0	2·8	3·5	—	—	—	—
n-Dodocane	—	—	—	12·8	8·5	6·5	—

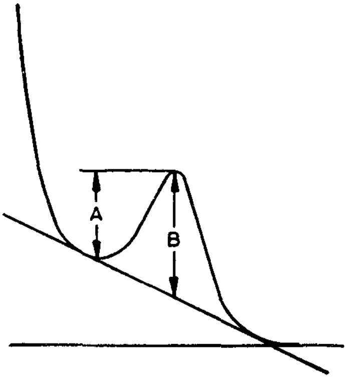

FIG. 2 ILLUSTRATION OF A/B RATIO FOR A SMALL COMPONENT PEAK ON THE TAIL OF A LARGE PEAK

4.3 Micro Syringe — 10 μl capacity, for sample introduction.

4.4 Volumetric Flasks

4.5 Volumetric Pipettes

4.6 Tubing — Copper, aluminium, or stainless steel.

4.7 Analytical Balance

5. REAGENTS AND MATERIALS

5.1 Liquid Phase for Column — *See* Note under **4.1.3.2.**

5.2 Solid Support — *See* Note under **4.1.3.2.** If an open tubular column is used, a solid support is not necessary.

5.3 Solvent for Liquid Phase — Reagent grade methylene chloride has been found satisfactory.

WARNING — Harmful if inhaled. High concentrations may cause death.

5.4 Carrier Gas — *See* Note under **4.1.3.2.**

CAUTION — Helium or nitrogen are compressed gases under high pressure.

5.5 Hydrogen — If a flame-ionization detector is used.

DANGER — Extremely flammable gas under pressure.

5.6 n-Hexane, n-Heptane, or iso-octane — 92 mole percent minimum containing less than 0·05 mole percent of aromatic material.

DANGER — Extremely flammable, harmful if inhaled, may produce nerve cell damage.

5.7 Benzene, Toluene, and Ethylbenzene — 99·5 mole percent minimum.

DANGER — Benzene is poison, carcinogen, harmful or fatal, if swallowed (*see* caution below).

CAUTION — Light aromatic hydrocarbons are toxic. Special precautions should be taken when handling such materials to avoid inhaling the vapour. If some of the liquid is accidentally spilled on the skin, the affected part should be washed immediately with soap and water.

5.8 *n*-Decane and Either *n*-Undecane or *n*-Dodecane — Either the C_{11} or C_{12} normal paraffin may be used as an internal standard according to the requirements in **4.1.3.**

CAUTION — Combustible, vapour harmful.

6. SAMPLE HANDLING

6.1 Due to the volatility of these materials and the tendency towards change in composition, store all samples and standard blends at a temperature of 7 to 15°C. For sampling at temperatures higher than 15°C keep the container open for minimum posssible time. The higher the temperature of sampling, the larger would be the errors.

7. CALIBRATION

7.1 Prepare a standard sample containing 0·5 percent by mass each of internal standard, benzene, toluene, and one of the C_8 aromatic compounds in *iso*-octane. Measure all the compounds by mass. To avoid evaporation loss weigh the compounds, starting with the heaviest compound, that is, in the reverse order to that listed above.

7.2 Convert from mass to liquid composition by dividing the mass of the comopound by the relative density of the compound as shown in Table 3. Calculate the concentration of the aromatic compound as percent by liquid volume to two decimal places.

7.3 Select the instrument conditions necessary to give the desired sensitivity (*see* Note 1 under **4.1.3.2**). Inject a known volume of the calibration blend in a quantity sufficient to meet the sensitivity requirements of the test. Change the attenuation, if necessary, so that all aromatic peaks are measured with a chart deflection of not less than 25 percent nor more than 95 percent of full scale for attenuated peaks. In deciding attenuation consider all unresolved peaks as a single compound. There may be tailing of the non-aromatic peak, but do not use any conditions such that the depth of the valley ahead of the benzene peak is less than 50 percent of the height of the benzene peak.

7.4 If there is tailing of the non-aromatic material, construct a baseline by drawing a line from the bottom of the valley ahead of the aromatic peak to the point of tangency after the peak (*see* Fig. 2). Measure the area of the aromatic peaks by any of the following means: traingulation, planimeter, paper cut-out, or mechnical or electronic integrator. Do not use integrators except on peaks having a constant baseline, unless the integrator has provision for making baseline corrections with accuracy comparable to or better than manual methods.

7.5 If a different attenuation was used for part of the chromatogram, correct to a constant attenuation basis by multiplying the area where a lower sensitivity was used by the ratio:

$$S_I/S_a$$

where

S_I = attenuatation sensitivity used for the internal standard peak; and

S_a = attenuation sensitivity used for the aromatic peak.

7.6 Relative Response Factors — Calculate relative response factors for each aromatic compound relative to that of the internal standard as follows:

$$R_a = (A_I/V_I) / (A_a/V_a)$$

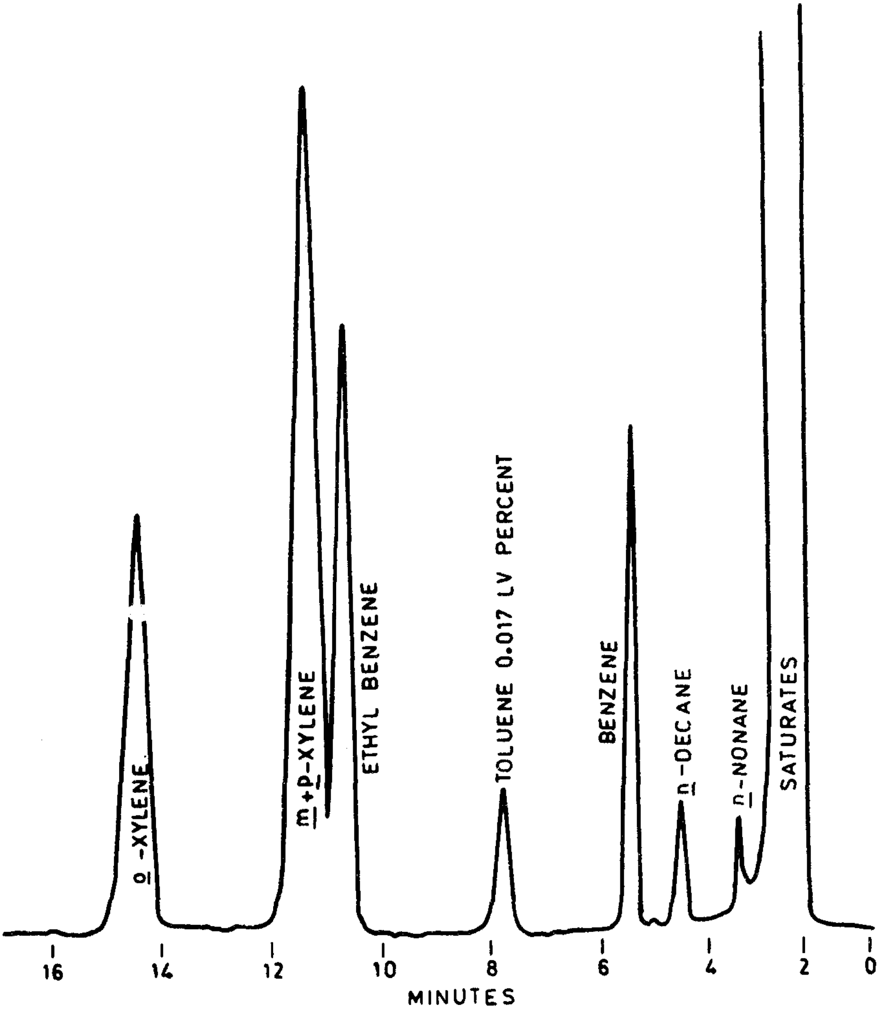

FIG. 3 TYPICAL CHROMATOGRAM (*see* Column 5, Table 1)

where

R_a = relative response factor for each aromatic compound;

A_I = area of the internal standard peak in arbitrary units corrected for attenuation;

V_I = volume of internal standard in the standard blend;

A_a = area of the aromatic peak in arbitary units corrected for the difference in attenuation; and

V_a = volume of aromatic in the standard.

8. PROCEDURE

8.1 Place approximately 0·1 ml of internal standard in a tared 25-ml volumetric flask. Determine the exact amount by weighing on an analytical balance. Fill to the 25 ml mark with sample and mix by inverting several times.

8.2 Calculate the volume percent of internal standard in the blend as follows:

$$V_I = [(M_I/S_I) / 25] 100$$

where

V_I = volume percent of internal standard in the blend;

M_I = net mass of internal standard; and

S_I = relative density 15·6°/15·6°C of the internal standard from Table 3.

TABLE 3 RELATIVE DENSITIES*

(*Clauses* 7.2 *and* 8.2)

COMPOUND	RELATIVE DENSITY OF 15·6°/15·6°C
(1)	(2)
Benzene	0·884 5
Toluene	0·871 9
Ethylbenzene	0·871 7
p-Xylene	0·865 7
m-Xylene	0·868 7
o-Xylene	0·884 8
iso-octane	0·696 3
n-Decane	0·734 1
n-Undecane	0·744 1
n-Dodecane	0·752 6

*Taken from Physical Constants of Hydrocarbons C_1 to C_{10}, ASTM DS 4; American Society for Testing Materials 1963.

8.3 Using the exact instrument conditions as were used in the calibration, inject the same volume of sample containing the internal standard. Change the attenuation if necessary so as to measure the area of the internal standard and aromatic peaks with not less than 25 percent chart deflection on attenuated peaks.

9. CALCULATIONS

9.1 Measure the area of the internal standard and aromatic peaks in the same manner as was used for calibration. If a different attenuation was used for part of the chromatogram, correct to a constant attenution basis by multiplying the area of the aromatic peaks by the ratio:

$$S_I/S_a$$

where

S_I = attenuation sensitivity used for the internal standard peaks; and

S_a = attenuation sensitivity used for the aromatic peak.

9.3 Calculate the concentration of benzene, toluene, C_8, and heavier aromatics as follows:

$$V = (A_a \times R_a \times V_I) / (A_I \times 0{\cdot}996)$$

where

V = volume percentage of the aromatic compounds;

A_a = area of the aromatic peak corrected to a constant attenution;

R_a = relative response factor for the aromatic compound;

V_I = volume percent of internal standard;

A_I = area of the internal standard peak; and

0·996 = factor to correct for the dilution by the internal standard.

Use the relative response factor for the C_8 aromatic in the calibration blend for all C_8 and heavier compounds.

9.3 Total Aromatics — Add the amounts of each aromatic compound found to obtain the volume percent of total aromatics in the sample.

9.4 Report — Report the volume percentage of each aromatic carbon number group and total aromatics to the nearest two significant figure, but not to more than three decimal places, for example, 0·86, 0·086, 0·009.

10. PRECISION

10.1 The criteria shown in Figs. 4 and 5 should be used for judging the acceptability of results (95 percent confidence).

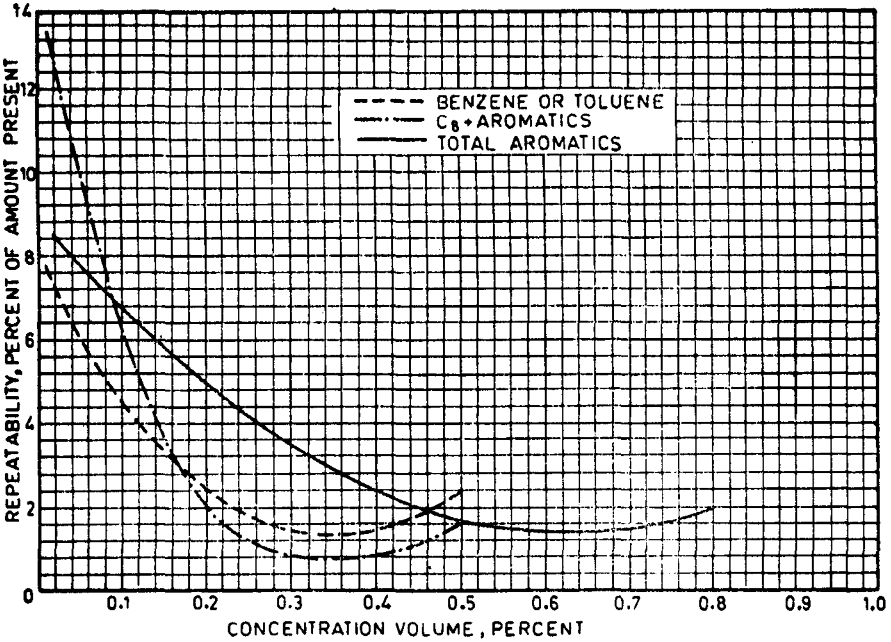

FIG. 4 REPEATABILITY

10.1.1 Repeatability — Duplicate results by the same operator should be considered suspect if they differ by more than the amount shown in Fig. 4 for repeatability as percent of the average amount present.

10.1.2 Reproducibility — The results submitted from two laboratories should be considered suspect if they differ by more than the amounts shown in Fig. 5 for reproducibility as percent of the average amount present (*see* Note).

NOTE — The columns, conditions and methods of area measurement used by the cooperators in developing the precision statement are shown in Table 1.

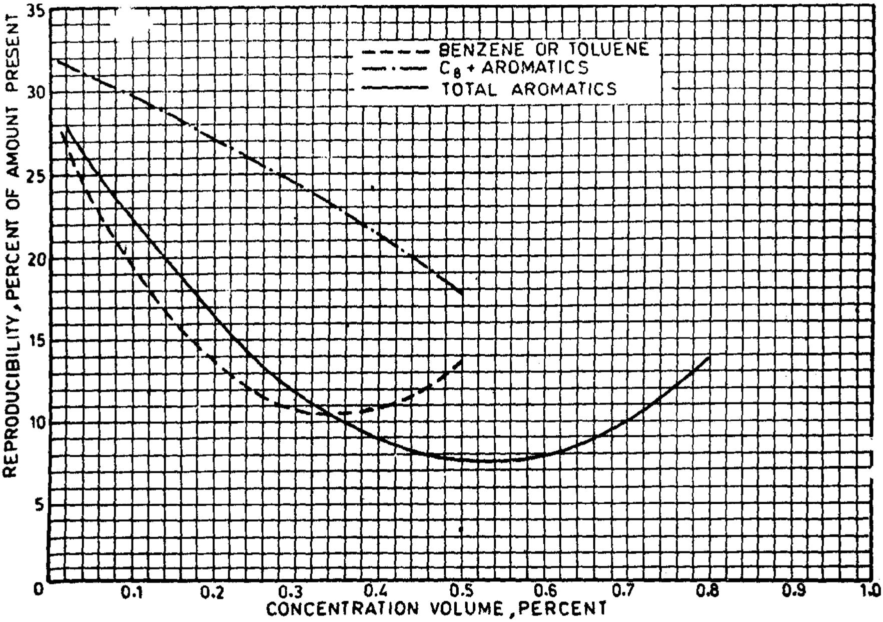

FIG. 5 REPRODUCIBILITY